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Measuring Odor Intensity with E-Noses and Other Sensor Types

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Abstract
This paper addresses the problem of predicting human olfactory ratings from instrumental measurements. Specifically, the paper analyzes the ability of two commercially-available electronic nose instruments (the NST 3320 and the Cyranose® 320) and three other sensing devices (a photoionization detector, a gold-film sulfur sensor, and an infrared laser detector) to match the performance of the human olfactory system in measuring odor intensity in a variety of realistic situations. These initial studies offer encouraging results for the two e-nose instruments, the PID, and the Au-film sensor.

Keywords
Odor intensity, odor measurement, photoionization detector.

INTRODUCTION
The prediction of human odor panel ratings from instrumental measurements is arguably the grand challenge of sensor-based machine olfaction. The complex transduction mechanisms and rich perceptual space of the human olfactory system pose serious sensory and computational questions for the development of a general-purpose instrument. The complexity of this machine-to-human mapping is clearly exemplified by the work of Burl et al. [1], who have attempted the prediction of organoleptic descriptors for a variety of odors in Dravnieks' atlas database using conducting polymer sensors. Correlation of sensory analysis with electronic nose data seems to be limited currently to specific odor domains. Some initial results along these lines have been reported in the literature, including beers [2], paperboard [3], packing materials [4], tomato paste and milk [5], indoor paints [6] and hog odors [7].

The present work addresses one of the dimensions of the human olfactory perceptual space: odor intensity. Measurement of odor intensity with an electronic nose can be a challenge due to sensitivity issues at low ppb levels as well as shifting baselines for the sensors. The purpose of this paper is to describe four experiments in which two E-noses (NST 3320, Applied Sensor, Parsippany, NJ, and Cyranose® 320, Cyrano Sciences, Pasadena, California) and three other sensor types (photoionization detector, gold film sensor, and infrared laser and detector) were used to measure odor intensity in a variety of applications. In each of the four experiments, the instrumental methods were compared to odor intensity data from a human odor panel.

EXPERIMENTS

Experiment 1: Effect of food additive on odor intensity of dog and cat feces

Twenty-five dogs and 43 cats were given food with 4 levels of a food additive intended to reduce the intensity of fecal odor. All dogs and cats first participated in a prefeed condition during which they consumed regular pet food. They then participated in one of 5 experimental treatment conditions: control (TC), treatment 1 (T1), treatment 2 (T2), treatment 3 (T3), and treatment 4 (T4). Treatments 1 to 4 were food containing increasing concentrations of the same additive. After a 3-day washout period the dogs then participated in a postfeed condition using regular food without the additive. Fecal samples were collected during a period when the animals were fed food with 1 of 4 levels of additive (or food with no additive), as well as before the treatment (prefeed), and after the treatment (postfeed). The samples were evaluated by both an electronic nose and a human sensory panel. The electronic nose used to evaluate the samples was the NST 3320 (Applied Sensor, Parsippany, NJ). This electronic nose has 22 sensors consisting of 10 MOSFET and 12 MOS types. These sensors respond to different classes of compounds and gases (see Table 1).

Multiple fecal samples were taken during each condition. A total of 450 dog fecal samples (from 25 different dogs) and 264 cat fecal samples (from 43 different cats) were evaluated by the human odor panel. A total of 451 dog fecal samples (from 25 different dogs) and 267 cat fecal samples (from 43 different cats) were evaluated by the electronic nose. Samples were frozen (-15 to -20 degrees C) until they were evaluated. Samples were allowed to thaw at room temperature (22 to 24 degrees C) for 2-3 hours before testing.

Samples were placed in glass bottles designed for the machine. The bottles were covered with Teflon-coated septa and plastic screw caps, also designed for use with this electronic nose. One gram of sample was placed in each glass bottle. The fecal samples were placed in the bottles in the order in which they would be tested, so that the samples would have approximately the same amount of time to build up equilibrium of headspace. Each sample, one by one, was warmed to 40 degrees C in an individual well in the machine for a 5-minute incubation phase, and after the 5-minute period, it was evaluated by the electronic nose. Each carousel well had its own

temperature control and was warmed independently of the other 11 wells. The order of presentation of the samples was randomized, with the exception that cat and dog samples were tested separately. The steady state of the sensor during exposure to the odorant minus the baseline (the steady state response of the sensor to reference air) was used for analysis.

The average ratings for the multiple samples of each dog were calculated for the different treatment conditions. The difference between the prefeed ratings and the experimental treatment ratings were then compared using a repeated measures model for the readings from the 22 different sensors. The results are shown in Table 1. For treatment 1, there was a significant change by treatment for 12 sensors: nine sensor values were lower for treatment 1, and 3 sensor values were higher for treatment 1. For treatment 2, there was a significant change by treatment for 11 sensors, with all 11 sensor values being lower for treatment 2. For treatment 3, there was a significant change for two sensors, and for treatment 4, there was a significant effect for 6 sensors.

The e-nose data were consistent with human data. For dog samples, there were significant reductions in nasal irritation for T2 and near significant reductions in odor intensity for T1 in humans. Both T1 and T2 had significant reductions for dogs in sensor responses on the e-nose. Neither the e-nose nor humans found measurably significant odor improvement by the additive for the cats.

Table 1. Compounds detected by individual sensors and effect of treatment for dog samples (Statistical significance is indicated in the table by *p<0.05, **p<0.005, ***p<0.001; the symbols ⇑ and ⇓ represent increases and decreases in sensor response, respectively)

Sensor	Important Detected Compounds	T1-pre	T2-pre	T3-pre	T4-pre
MOSFET 101A	Hydrogen, amine	⇓*	-	-	-
MOSFET 101B	Amine, aldehyde, ester, alcohol, ketone	⇓*	-	-	-
MOSFET 102A	Amine, ester	⇓**	⇓*	-	-
MOSFET 102B	Hydrogen, amine, alcohol	-	-	-	-
MOSFET 103A	Amine, aldehyde, alcohol	-	-	-	-
MOSFET 103B	Amine, aromate, aldehyde, ester, alcohol, ketone	⇓*	⇓*	-	-
MOSFET 104A	Hydrogen	⇓*	-	-	-
MOSFET 104B	Hydrogen	⇓*	⇓*	⇓*	-
MOSFET 105A	Hydrogen, amine	⇓***	⇓***	-	-
MOSFET 105B	Hydrogen, amine, aldehyde, ester, alcohol, ketone	-	-	-	-
MOS 101	Air contaminants (hydrogen, carbon monoxide)	-	-	-	⇓*
MOS 102	Hydrocarbons	-	⇓***	-	-
MOS 104	Alcohol, organic solvent	-	⇓*	-	⇓*
MOS 110	Hydrocarbons	⇑**	-	⇑*	-
MOS 111	Methane	⇑**	-	-	-
MOS 112	Propane, butane	-	⇓*	-	⇓*
MOS 113	Hydrogen	⇑*	-	-	⇓*
MOS 114	Organic solvents	-	⇓**	-	-
MOS 115	Alcohol	⇓***	-	-	-
MOS 116	Freon	⇓*	⇓**	-	-
MOS 117	Ammonia	-	⇓*	-	⇓*
MOS 118	Organic solvents, alcohol, hydrogen	-	⇓**	-	⇓*

Experiment 2:
Relationship of odor intensity to particle size for particulates emitted from a swine facility

The air emitted from fans at a swine facility was sampled with an Andersen 8 Stage Non-Viable Cascade Impactor. Classification stages of the impactor consisted of eight jet plates and impaction discs that can classify aerosols from 9 micrometers and above to 0.4 micrometers (at 28.3 lpm). Odors from the entrained particles that settled upon the glass filters mounted on the impaction surfaces of each stage were sniffed by a human panel and by two electronic noses (the NST 3320 and the Cyranose® 320). The human panel and both e-noses were able to rank order the intensity of the odors by particle size with smaller particles having greater odor intensity (see Table 2 and Figure 1). A plain glass filter (labeled D) was used as a reference in Figure 1.

Table 2. Odor intensity data versus particle size

Stage	Intensity (std error)
0	1.878 (0.460)
1	2.192 (0.396)
2	2.432 (0.509)
3	2.326 (0.470)
4	2.507 (0.547)
5	2.571 (0.566)
6	2.652 (0.616)
7	2.887 (0.613)

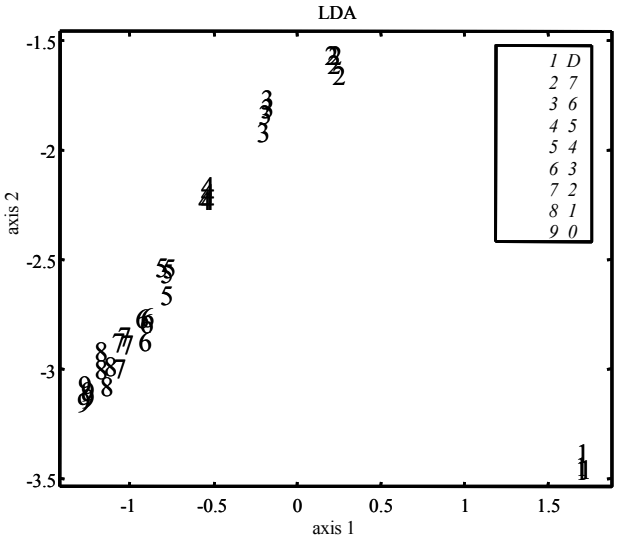

Figure 1. Analysis of NST enose data.

Experiment 3:
Assessment of odor intensity and VOC levels of indoor and outdoor air

The purpose of this experiment was to investigate the feasibility of measuring odor intensity with a new portable machine that is designed for real-time monitoring of volatile organic compounds (VOCs) at ppb levels (ppbRAE VOC Monitor PGM-7240 from RAE Systems, 1339 Moffett Park Drive, Sunnyvale, CA 94089). The sensor is a photo-ionization detector (PID) that can detect VOC concentrations down to a few ppb and is minimally sensitive to interference from humidity. The PID determines the concentrations of gases including organo-heteroatom species such as organosulfur or organophosphorus species by using ultraviolet (UV) light to ionize the analyte. When the energy of an incoming photon is high enough, an electron can be temporarily removed from its molecular orbital leading to ionization, i.e. $R + h\nu \rightarrow R^+ + e^-$. The ions produced by this process are collected by electrodes, and a current is generated that is proportional to the analyte concentration. The PID is considered a nondestructive detector because only a very small fraction of the analyte molecules are actually ionized in its chamber. The ppbRAE utilizes a 10.6eV lamp; thus the energy is adequate to ionize compounds with ionization potentials (IP) less than 10.6 eV. The IP of most nonodorous atmospheric gases such as nitrogen, oxygen, and carbon monoxide are greater than 12.0 eV, while organic compounds generally have ionization potentials less than 12.0 eV.

In this study, simultaneous measurements of odor and VOC levels (using the ppbRAE) were compared. A trained odor panel evaluated the odor intensity of a wide variety of indoor and outdoor environments. Perceived odor intensity was measured on a 9-point scale, from 0 (none at all) to 8 (maximal) where 0 was "none at all"; 1 was "very weak"; 2 was "weak"; 3 was "moderate weak"; 4 was "moderate"; 5 was "moderate strong"; 6 was "strong"; 7 was "very strong"; and 8 was "maximal". At the same time, the ppbRAE was used to determine the total concentration of VOCs at the same location. The locations that were evaluated included: multiple stores in two different shopping malls, each room of two different houses, locations in the Taste and Smell Laboratory and other places at Duke University Medical Center and Duke campus, coffee shops, and inside cars. Table 3 gives examples of locations tested and odor intensity perceived by the human panel. Figure 2 shows the relationship between the mean VOC levels measured by the ppbRAE and perceived odor intensity at incremental levels from 1 to 5. The regression equation is $y = 37.861 + 49.693x + 2.7294x^2$ and the Pearson correlation coefficient between odor intensity and VOCs as measured by the ppbRAE is 0.914.

Experiment 4:
Assessment of odor intensity and VOC levels near swine operations

A final experiment was undertaken to compare odor intensity ratings at swine facilities determined by the human panel with measurements of VOC levels (using the ppbRAE), sulfurous compounds such as hydrogen sulfide (using the Jerome meter, Arizona Instruments), and concentrations of particulates 10 μm and smaller (using the Haz-Dust EPAM5000, Environmental Devices Corporation.) The Jerome meter uses a gold film sensor that is selective to hydrogen sulfide (sensitive to 1 ppb) resulting in a change in electrical resistance (potential interference from ammonia but not SO_2, CO_2, CO and water vapors). The EPAM uses an infrared laser and detector such that airborne particles that carry odorants scatter light from an infrared laser. VOCs, hydrogen sulfide, and particulates were selected for measurement because they are among the predominant emissions experienced downwind by neighbors of large swine facilities. Instrumental measurement of emissions is necessary to document exposures when trained human assessors from control agencies are unavailable to monitor odor levels. Table 4 provides a summary of results from this second study. Results were averaged for three different odor intensity ranges: from 1 - 4, from 4.5 - 6, and from 6.5 - 7 on the odor scale described above.

Table 3. Results from Experiment 3.

Location	Odor intensity	Location	Odor intensity	Location	Odor intensity
Inside mall parking deck	3	clothing store A	1.5	calendar store	2
near edge of mall parking deck	2	clothing store B	2.5	leather store A	2.5
next to mall parking deck	1.5	chicken sandwich shop	3.5	leather store B	3
inside cigar shop humidor	5	perfumed lotion store	3.5	leather store C	4
mall area near restaurant	1.5	doughnut shop	3.5	department store	2.5
gift shop, near potpourri	4.5	gift card store	3.5	inside elevator	1.5
in mall between yogurt & cookie shops	3	food court area in mall	4	bedroom in house	1
luggage and gift store	2	food & cooking store A	2.5	photography shop	4.5
outside air on Duke campus	1	food & cooking store B	4	candle store A	4.5
inside campus center	2	coffee shop A	4	candle store B	5
inside our laboratory	1	coffee shop B	4.5	inside library	1
headspace of dog feces samples	5.5				

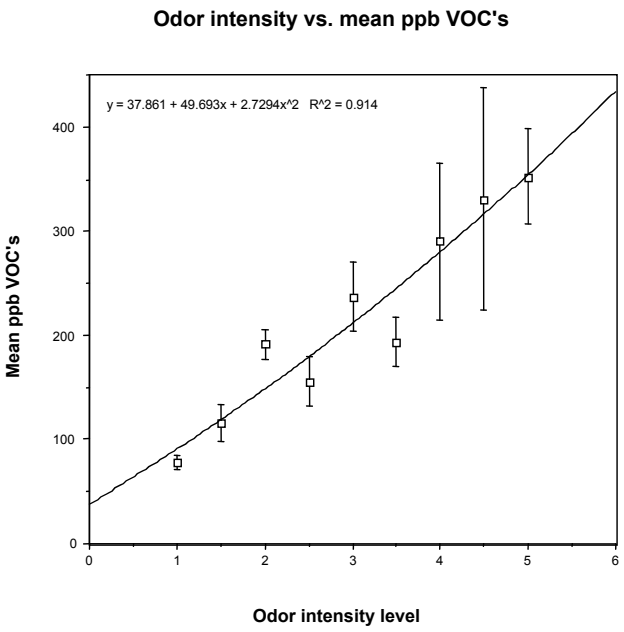

Figure 2. Correlation of human panel and ppbRAE measurements for Experiment 3

Table 4. Relationship of odor intensity to instrumental ratings in Experiment 4

Perceived odor intensity range		(1 - 4)	(4.5 - 6)	(6.5 - 7)
VOC's (ppb)	average	3.8	15.3	38.0
	maximum	8.8	66.0	116.0
H_2S (ppb)	average	5.9	16.5	51.7
	maximum	7.3	42.5	79. 7
particulates (mg/m^3)	average	0.030	0.017	0.040
	maximum	0.071	0.028	0.073

The results from this experiment indicate that concentrations of VOCs (as measured by the ppbRAE) and H_2S (as measured by the gold film sensor) but not particulates correlated with odor intensity as judged by a trained odor panel.

CONCLUSIONS

This paper has presented an evaluation of five odor sensing instruments for the purpose of predicting human odor-intensity ratings. Responses from two e-noses (Applied Sensor NST 3320 and Cyranose® 320) and two other sensing devices (photoionization detector and gold film sensor) correlated to some degree with odor intensity in a variety of applications.

In the first of the experiments in this article, the e-nose provided results consistent with human olfactory ratings. Both machine and human data showed significant or near-significant changes for two of the four dog-food treatments, and no significant changes in cat foods. The second experiment provided conclusive results showing a strong correlation between perceived odor intensity, particle size and e-nose patterns. A photoionization detector was used in the third set of experiments to collect data from a variety of locations. Human intensity ratings and instrument data showed a Pearson correlation coefficient of 0.914. The final experiment measured emission levels at swine operations using the PID, a gold-film sensing device and an infrared laser detector. Data from the PID and Au-film sensor, but not from the laser detector, correlated with odor intensities from an odor panel. More research is needed, however, to develop better instrumental methods that provide accurate odor intensity values that reflect human perception.

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